SYNTHESIS AND CHARACTERIZATION OF ULTRA VIOLET CURABLE RENEWABLE POLYMER GRAPHITE COMPOSITES

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A thesis submitted in
fulfillment of the requirement for the award of the
Doctor of Philosophy

Faculty of Mechanical and Manufacturing Engineering
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JULY, 2019

DEDICATION

THE BELOVED PERSON

My late father

HJ ABDULLAH BIN ASMAWI

Al-Fatihah

The rest of my family member

HJH SAPIAH BINTI TOMPANG

HAFIZAH BINTI ABDULLAH

NOORIZAN BINTI ABDULLAH

MOHD RIZWAN BIN ABDULLAH

MUHAMMAD NUR HAFIZ BIN ABDULLAH

MOHD FAIZ BIN AHAT

ACKNOWLEDGEMENT

In the name of ALLAH, The Most Compassionate, The Most Merciful. Salawat and Salam to our beloved Prophet, Nabi Muhammad S.A.W. For the past 6 years, I have been supported by a great number of people and it is a pleasure to convey my gratitude to them all in my acknowledgments.

First of all, I would like to express million thanks to my supervisor, Prof. Dr Anika Zafiah binti Mohd Rus and my co-supervisor, Prof. Dr Mohammad Faiz Liew bin Abdullah, whose enthusiasm, critical thinking and optimism inspired me to embrace research in every possible way. By sharing their expertise, I have been given a great chance to experience and improve efficiently through attending conferences, exhibitions and exposure to impact publications. Their guidance truly inspired and enriched my growth as a good teacher and researcher that I want to be. I am also grateful to my current and former colleagues, and technicians in Sustainable Polymer Engineering Group (SPEN- AMMC), Faculty of Mechanical and Manufacturing (FKMP) and MiNT-SRC FKEE, UTHM for their assistance, discussions and suggestions. May the 'silaturrahim' last forever.

I owe my deepest gratitude to my family. Their unconditional love, overwhelming support, and encouragement have allowed me to pursue my successes. I can say "my family is my life" and I have always wanted to be your pride.

Last but not least, I would like to thank everybody who was important directly and indirectly to the successful realization of this thesis, as well as expressing my apology that I could not mention personally one by one. May Allah give His blessing to all of us. Aamin

~I am indebted to them more than they know~

ABSTRACT

This thesis aims is to evaluate the synthesis and characterization of ultra violet (UV) curable renewable polymer graphite (RPG) composites. Accordingly, the renewable polymeric composites were prepared through a film slip casting method at room temperature wherein graphite particles of various weight loadings were mixed with mass proportion 2:1 of renewable monomer and Methylene Diphenyl Diisocyanate, MDI respectively. The main concerned was given to renewable monomer based vegetable cooking oil produced at the SPEN-AMMC UTHM. The morphology-structural relation of the RPG composites confirmed that the graphite particles contain functional groups such as hydroxyl and carboxylic groups are randomly distributed and attributed to formation of interconnected interface within the polymeric composites. Furthermore, as the graphite particle loading increased, the thermal degradation temperature at three distinct decomposition stages shifted and to some extent, resulting in much higher crystallinity. As expected, the mechanical properties of the composites were also enhanced with the modulus and tensile strength increment up to ~440% and ~100% respectively. Significantly, all of these results correlate with viscoelastic properties in which the composites achieved percolation threshold at RPG_{20} composites. Moreover, the decreased in optical energy band gap (E_g) which afterwards took the leads to electrical conductivity (σ). Aptly, the composites ($RPG_{20,}$ RPG_{25} and RPG_{30}) were found to possess favorable electrical conductivity range of $10^{-5} – 10^{-4}$ S/m, while all other samples were deemed to be not conductive due to improper dispersion of graphite particulates. On the contrary, UV curable composites did not show any significant enhancement and graphite particle acted as UV stabilizer in this manner. Therefore, the stability of the conductive renewable polymer graphite composite is suitable to be used in various composites applications.

ABSTRAK

Tesis ini bertujuan untuk menilai sintesis dan pencirian komposit polimer grafit (RPG) yang boleh diperbaharui melalui terapi ultra violet (UV). Oleh itu, komposit polimer boleh diperbaharui telah disediakan melalui kaedah gelincir pemutus filem pada suhu bilik di mana zarah grafit pelbagai berat beban dicampur dengan nisbah 2: 1 antara monomer boleh diperbaharui dan Methylene Diphenyl Diisocyanate, MDI. Keutamaan diberikan kepada penghasilan monomer boleh diperbaharui daripada minyak masak sayur di SPEN-AMMC UTHM. Hubungkait morfologi-struktur komposit RPG mengesahkan bahawa zarah grafit mengandungi kumpulan berfungsi seperti kumpulan hidrosil and karboksil yang diserakkan secara rawak serta menyumbang kepada pembentukkan ikatan dalam komposit polimer tersebut. Tambahan pula, seiring dengan peningkatan zarah grafit, suhu kemerosotan termal pada tiga peringkat penguraian yang berbeza beralih dan menyebabkan penghabluran bahan yang lebih tinggi. Dijangkakan, sifat mekanik komposit juga dipertingkatkan dengan kenaikan kekuatan modulus dan tegangan masing-masing sehingga ~ 440% dan ~ 100% . Nyata, keseluruhan keputusan ini bersesuaian dengan sifat viskoelastik di mana komposit mencapai puncak perkolasi pada 20% beban grafit (RPG_{20}). Selain itu, penurunan dalam jurang jalur tenaga optik (E_g) yang kemudiannya membawa petunjuk kepada kekonduksian elektrik (σ). Seterusnya, komposit (RPG_{20}, RPG_{25} dan RPG_{30}) didapati mempunyai rangkaian kekonduksian elektrik yang baik iaitu $10^{-5} – 10^{-4}$ S/m, manakala semua sampel lain dianggap tidak konduktif disebabkan penyebaran sebatian grafit yang tidak sekata. Sebaliknya, komposit yang melalui terapi UV tidak menunjukkan peningkatan yang signifikan dan zarah grafit bertindak sebagai penstabil UV dengan kajian ini. Oleh itu, kestabilan komposit grafit polimer konduktif sesuai untuk digunakan dalam pelbagai aplikasi komposit.

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LIST OF SYMBOLS AND ABBREVIATIONS

SPEN-AMMC	-	Sustainable Polymer Engineering, Advance Manufacturing and Material Centre
ASTM	-	American Society for Testing Materials
CPCs	-	Conducting polymer composites
RPG	-	Renewable polymer graphite
VCO	-	Virgin cooking oil
UV	-	Ultra violet
H_2SO_4	-	Sulfuric acid
H_3PO_4	-	Orthophosporic acid
H_2O_2	-	Hydrogen peroxide
µ	-	Mikro
R	-	Carbonyl hydroxyl index
Xc	-	Crystallinity
m	-	Mass
ρ	-	Density
m	-	Mass
µ	-	Mikro
R	-	Carbonyl hydroxyl index
E’	-	Storage modulus
Ve	-	Cross-link density
n	-	Refractive index
k	-	Extinction coefficient
α	-	Absorption coefficient
I-V	-	Current-voltage
ρ	-	Electrical resistivity
σ	-	Electrical conductivity

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CHAPTER 1

INTRODUCTION

1.1 Introduction and motivation

> “We are used to the great impact scientific discoveries have on our ways of thinking. This year's Nobel Prize in Chemistry is no exception. What we have been taught about plastic is that it is a good insulator - otherwise we should not use it as insulation in electric wires. But now the time has come when we have to change our views. Plastic can indeed, under certain circumstances, be made to behave very like a metal”
>
> Heeger, MacDiarmid, & Shirakawa, the Nobel Prize in Chemistry 2000.

Adding conductive fillers to the polymer matrix yields the composites that are also conductive, and these composites have many applications including electromagnetic shielding (Al-Saleh, 2016), solar cell (Wang, 2013), wearable piezoresistive sensor (He *et al*., 2017), coatings (Riley *et al*., 2016) and energy storage (Pielichowska *et al*., 2016). Therefore, these discoveries of conducting polymeric composites (CPCs) have marked the beginning of a completely new field (Heeger *et al*., 2000). The CPCs possess electronic conductivity similar to those metals or behave like organic semiconductors. Considering the vast amount of new possibilities the conductive polymers offer, it is expected that the CPCs would find numerous applications across various industries such as in electronics, automobiles, healthcare and heavy industry.

Generally, polymeric materials constitute π conjugation in their backbone, and responsible for the phenomena of delocalization. The delocalized π electrons generate conductivity throughout the polymer chain via the formation of polarons and bipolarons. Their conductivity can be further increased in several orders by means of doping (Balint *et al*., 2014). Among various insulating polymers matrices are like those of epoxy, alkyl, polyacrylates, PU etc., likewise, PU is the most important one which extensively used and of versatile nature, comprises hard (isocyanate) and soft (polyols) segments (Zhang *et al*., 2017). Its end product properties can be designed according to user needs. Nonetheless, under harsh conditions PU by itself fails to give satisfactory thermal, mechanical and electrical performance (Almeida Júnior, 2013; Plyushch *et al*., 2016; Wu *et al*., 2018). Hence, there is a need to induce such structural properties in PUs through their blending and composites formations, which enable them to provide good services even under harsh environmental conditions. It has been observed that the synergistic effect between carbon-based (filler) and insulating PUs (matrices) enhances their proccessability, stability, solubility, thermal, mechanical, electrical and optical properties (Sattar *et al*., 2014).

Moreover, carbonaceous nanofillers such as graphite, graphene and carbon nanotubes (CNTs) play a very promising role for the reason of their better structural and functional properties such as high aspect ratio, high mechanical strength high electrical properties etc. than others (Rony, 2015). Principally, graphene is the mother element of the carbon allotropes. In 2004, two scientists from Manchester University, Andre and Kostya, removed some flakes from a lump of bulk graphite with sticky tape and discovered that some flakes were thinner than others. Inspired by this particular event, they figured out how to create one atomic thickness. The playful, yet groundbreaking, approach has eventually led scientists to the 2010 Nobel Prize in Physics (Hancock, 2011). As a result, there has been an enormous amount of research being conducted to integrate graphene into numerous applications. The field of graphene, related 2d crystals and hybrids is now rapidly developing from pure science to technology. The current and near future market for graphene related materials applications is driven by the production strategies for these materials. In view of that, once each production route is mature enough, this will facilitate a widespread practical implementation of these

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